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DEVELOPMENT OF LIQUID CRYSTAL LAYER THICKNESS AND REFRACTIVE INDEX MEASUREMENT METHODS FOR SCATTERING TYPE LIQUID CRYSTAL DISPLAYS

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We report the measuring method of scattering type display liquid crystal layer thickness based on capacitance values suitable for inline production process control. The method is selected for its effectiveness and simplicity over spectroscopic methods as conventional methods for scattering type displays are not applicable. During the method approbation process, a novel diffuser liquid crystal mixture refractive index was determined based on liquid crystal layer thickness measurement data.

Keywords: *Cell gap, COMSOL, fast switching diffusers, LCD.*

1. INTRODUCTION

Liquid crystal (LC) layer thickness (commonly known as "cell gap") determination is one of the most critical measurements in liquid crystal display (LCD) development and manufacturing, since it determines their most important optical properties, such as transmission and scattering.

In LCD production process, liquid crystal layer thickness between both sides of display glass is formed during vacuum assembly. Thickness is controlled by adjusting spacer size and liquid crystal volume inside the cell. There are deviations caused by liquid crystal dispensing process and evaporation rate in vacuum during assembly process which decrease production yield or lead to variations in optical properties. Excess of LC can cause visually distinct regions in lower part of display during intense use, called "gravity mura" [1].

Methods for determination of liquid crystal layer thickness using light polarization are well developed and commercially available but do not work for diffuser (light scattering) type LCDs [2]. Scattering LC displays become more and more important in times of digitalization, since they allow for fast adaptive optical elements and high-quality AR/VR visualization displays and glasses [3].

It has been shown that by using mathematical methods a cell gap can be calculated from transmittance spectrum [4], [5]. This indirect measurement method has drawbacks as it is done offline after the first sample production and requires idle production time while waiting for all process steps to be completed. For longer production runs process drift can happen without detection, even with periodic sample check. For certain cases, the use of interference patterns formed by the quasi-monochromatic light reflected from LCD has been reported for inline quality process control [2].

Second, it is not precise. Latest developments in diffuser technology have led to the situation when refractive indices (RI) of various layers are closely index matched, so spectroscopic determination of the maximum peaks becomes difficult. Moreover, this requires RI of material in the layer to be known. However, if novel LC mixtures are used, RI measurement requires to know layer thickness, first. LC refraction index over spectra measurement can be done by multi-wavelength Abbe refractometer [6] or using the spectral reflectometry method [7]. The spectrometer processes the light spectrum reflected or permeated by the sample. If the sample consists of a multilayer system in which multiple internal reflections occur, the interference bands may be observed in the spectrum, whose position is determined by the optical properties of the investigated layers – thickness and refractive index. At the Institute of Solid State Physics, this method is used to determine the breaking coefficients of 1 μm thick layers [5]. Abbe refractometer method has a problem in accuracy because it is difficult to control alignment distribution of LC.

If gap (layer)is filled by air instead of LC material, the interference peak determination is easy but it is commonly known in the industry that cell gap for unfilled displays is larger than for filled displays. After the layer is capillary-vacuum filled, extra pressing step is required to planarize the glass substrates and to bring filled layer to final value, when glass is resting on cell gap spacers. Thus, the layer thickness measured for unfilled displays is not valid any more. For more modern manufacturing method "one drop fill" display liquid crystal layer thickness is directly formed in vacuum with LC drops present, so the layer thickness is dependent on dispensed LC volume.

To overcome these limitations, scanning white light interferometric method was proposed [6]. While capable of determining liquid crystal layer thickness of different display types, it is not yet tested for scattering type of displays.

Additional challenge is the requirement to measure diffuser LCD in transparent mode after all production steps have been finished, as scattering mode typically hinders spectroscopic determination.

Capacitance measurements on the contrary can more easily be integrated in the production process as it is more simple technique. Display capacitance is dependent of liquid crystal layer thickness, and using its measurement we can compare one display to another in the same series on condition that other parameters are constant and controlled. It might provide a convenient method for in-line process control, and if exact dependence is known, even absolute liquid crystal layer thickness values can be obtained. Display modelling by finite elements method can provide values of capacitance dependence from liquid crystal layer thickness, so a precise model of capacitive and resistive structures (Fig. 2) must be built. COMSOL Multiphysics software frequently is used for modelling complex capacitive systems in displays [8]. Use of software allows modelling displays with more complex electrode geometries like interplane switching electrodes or raised (slanted) electrode regions.

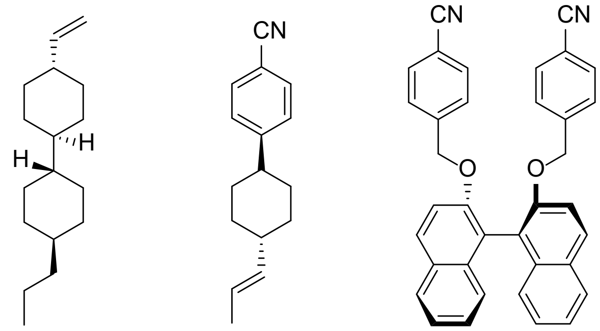
2. EXPERIMENTAL

Two types of functional LCD displays (61.8 x 48.8 mm) were prepared for the study using 2.7 µm spacers and the same layer construction but filled with different LC:

- Most commonly used standard “E7” LC mixture with well-known properties for reference purposes;
- Diffuser LC for measurement.

According to each LC type, polyimide layer had different LC alignment properties, planar for “E7” cell and homeotropic for diffuser.

Diffuser LC was prepared from mixture of “E7” (supplied by Merck) and 3 more components (supplied by the Latvian Institute of Organic Synthesis (LIOS)) with unknown refraction index, see Fig. 1.

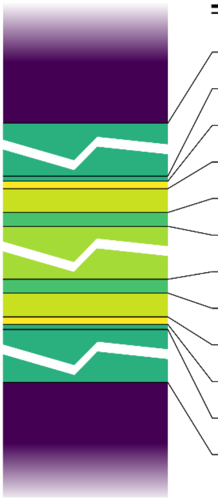


1 **2** **3**

Fig. 1. Chemical compounds synthesized by LIOS, added to LC mixture. **1** ((1s,1’r,4R,4’R)-4-propyl-4’-vinyl-1,1’-bi(cyclohexane)), **2** (4-((1s,4s)-4-((E)-prop-1-en-1-yl)cyclohexyl)benzonitrile), chiral dopant **3** ((S)-4,4’-(([1,1’-binaphthalene]-2,2’-diylbis(oxy))bis(methylene))dibenzonitrile).

MODELLING AND VALIDATION

In order to evaluate liquid crystal layer thickness from measured capacitance data, diffuser and “E”7 display capacitive structure simulation model was built using COMSOL Multiphysics software, see Fig. 2. The model was used to analyse capacitance dependence on liquid crystal layer thickness, see Table 1.



Material	Thickness d, nm	Refractive Index n	Relative dielectric permittivity
Air	-	1.00	1.0
Glass	550000	1.52	7.75
SiO_2 barrier layer	20	1.52	2.18
ITO conductive layer	30	1.82	-
Silica based hard coat	95	1.75	14
PI alignment layer	55	1.58	3.6
LC layer	8000	1.71	9.6
PI alignment layer	55	1.58	3.6
Silica based hard coat	95	1.75	14
ITO conductive layer	30	1.82	-
SiO_2 barrier layer	20	1.52	2.18
Glass	550000	1.52	7.75
Air	-	1.00	1.0

Fig. 2. Capacitative and resistive structure of diffuser. Refractive index and permittivity data from supplier data sheets. On the left, glass and LC layer thickness is not shown on scale, indicated by zig-zag white lines.

Table 1. Modelled Capacitance Values

LC thickness, μm	Diffuser display capacitance, nF	"E7" display capacitance, nF
2.8	74.2	148.2
2.9	72.1	144.1
3.0	70.2	140.3
3.1	68.3	136.6
3.2	66.5	133.1

CAPACITANCE MEASUREMENTS

Both display types were measured for capacitance values with Agilent multimeter model 3606A and compared to modelled results, see Table 2. Measured values are lower than the modelled results. Multimeter uses lower voltage (20V) well below a switching threshold, when we are not completely aligning LC molecules and their orientation is unknown, so average relative permittivity is unknown. Note higher liquid crystal layer thickness value for sample P8-6, as it comes from different (assembled) LCD panel.

There are two other methods of capacitance measurement that can be used, one with resistor in series (method "R"), see Fig. 5, and second (method "C") with capacitor in series instead of resistor.

Table 2. Capacitance Values of Tested Displays with E7

Sample ID	Calculated gap, μm	Capacitance modelled, nF	Capacitance, Agilent 3606A, nF	Capacitance, method "R", nF	Capacitance, method "C", nF
P8-6	3.3	148.2	85±1	129±1	131±0.6
P6-13-R	2.9	148.2	78±1	144±1	144.1±0.6
P6-24-R	2.8	148.2	74±1	152±1	152.8±0.6
P6-6-R	3.0	148.2	78±1	139±1	140.1±0.6
P6-11	2.8	148.2	72±1	147±1	145.5±0.6

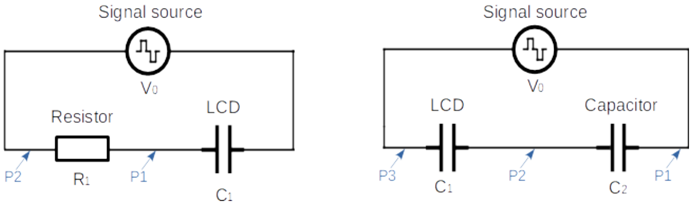


Fig. 5. Capacitance measurement methods. On the left with resistor in series "R", on the right with capacitor in series "C".

The test setup can be simplified to an RC circuit with a diffuser acting like a capacitor. The probes of an oscilloscope are connected to a resistor with a known and fixed value of approximately 10 k ohms. Also, the oscilloscope is configured to capture a measured signal when its value crosses some specific voltage level. For this experiment the trigger value can be set up to +10 and -10 volts for detecting the charge and discharge voltage curves, respectively.

From a standard RC circuit analysis, it is known that the voltage across the resistor will alter only during some rapid changes of the supply voltage or in our case, when the power supply is connected to and disconnected from the circuit. The voltage change on the resistor can be recorded and then converted to a current:

$$I(t)=V_m(t)/R\,, \tag{1}$$

which, by integrating it over the time, is transformed to the total charge that was accumulated on or released from the capacitor.

$$q=\int_{T_{START}}^{T_{FINISH}} I(t)dt\,. \tag{2}$$

This charge is then transformed to a capacitance:

$$C=q/V_s\,, \tag{3}$$

where V_s is the supply voltage.

The method with capacitor in series "C" is suggested [8] for faster switching systems, so it is preferred for this case. The method is also more convenient as capacitance value can be calculated directly from voltage measurements:

$$C_1=C_2\frac{V_2}{V_1}\,, \tag{4}$$

where
C_1 – capacitance of the diffuser;
C_2 – capacitance of serial capacitor that is known (300 nF for E7 cells);
V_1 – the voltage on capacitor C_1;
V_2 – the voltage on capacitor C_2.

Using method "C" measurements with capacitor with known value in series, we get similar capacitance results as with method "R".

SPECTROSCOPIC DETERMINATION

"E7" filled displays were also measured by a spectroscopic method. The required spectra in the 400–700 nm range were obtained using the Agilent Cary 7000 UMS spectrophotometer in five locations of each cell and liquid crystal layer thickness was calculated using the method developed by the Institute of Solid State Physics. The following parameters were used:

- Range ~550-750;
- No of points: 5;
- No of smooth cycles: 2;
- Fixed RI: 1.

Optical anisotropy of E7 LC is n_e=1.7472 and n_o=1.5217.

Extraordinary refractive index was used to calculate liquid crystal layer thickness (see Table 3). There is no difference observed when voltage is applied for most of the samples as expected since LC rotates only in XY plane.

Table 3. **Liquid Crystal Layer Thickness of LCD Samples with E7**

Driving conditions	Voltage on, 5V		Voltage off, 0V		Data from capacitance studies
Sample ID	LC layer thickness, µm	Stdev	LC layer thickness, µm	Stdev	LC layer thickness, µm
P8-6	3.0	0.2	3.0	0.2	3.3
P6-13-R	2.8	0.4	2.8	0.6	2.9
P6-24-R	2.9	0.2	2.9	0.2	2.8
P6-6-R	3.1	0.1	3.1	0.3	3.0
P6-11	3.1	0.2	3.1	0.2	2.8

REFRACTIVE INDEX DETERMINATION

To use a spectroscopic method for further cell gap measurement of diffuser displays, a refractive index must be known. To determine it, the layer thickness of LC must be known, first. If we take precautions that displays produced in the same conditions have the LC layer thickness, and fill some of them with liquid with the known refractive index different from adjacent boundaries so that interference peaks can be detected with ease and precision in transmission spectra. Obvious choice would be to use isotropic materials instead of LC, such as ethylene glycol and cinnamaldehyde. However, these materials evaporate during filling process in vacuum chamber, so LC with the known refraction index, "E7" typically used for comparable studies, was selected.

Displays were prepared in one batch, so one assumes that all test displays have equal LC layer thickness but an unknown absolute value, somewhat different from spacer size of 2.7 µm. To ensure equal layer thickness, during end sealing process when the excess of LC is squeezed out from cells under press, displays were mixed, see Fig. 4.

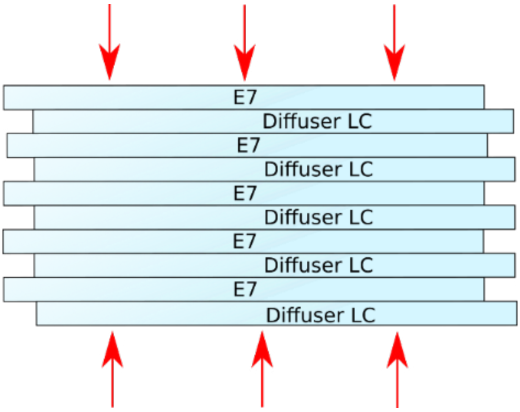


Fig. 4. Display pressing during end seal press step.

Using LC layer thickness calculated for samples with E7 LC, refractive index of diffuser LC was calculated and average *n* calculated, see Table 4. See values plotted in Fig. 5.

Table 4. Refractive Indices of Diffuser LC

Wavelength, nm	P5-6-R	P5-20-R	P5-15-R	P5-16-R	P5-10	P5-19	Average, n
738	1.52	1.48	1.50	1.50	1.50	1.48	1.50
711	1.52	1.51	1.51	1.50	1.51	1.51	1.51
683	1.53	1.55	1.52	1.57	1.52	1.52	1.53
660	1.57	1.55	1.55	1.57	1.55	1.52	1.55
636	1.57	1.51	1.55	1.58	1.55	1.55	1.55
617	1.60	1.58	1.59	1.61	1.54	1.58	1.58
596	1.68	1.61	1.61	1.64	1.61	1.57	1.62
580	1.70	1.62	1.63	1.71	1.62	1.62	1.65
563	1.78	1.69	1.69	1.72	1.63	1.69	1.70
549	1.69	1.66	1.67	1.70	1.72	1.66	1.68
535	1.81	1.77	1.77	1.81	1.77	1.69	1.77

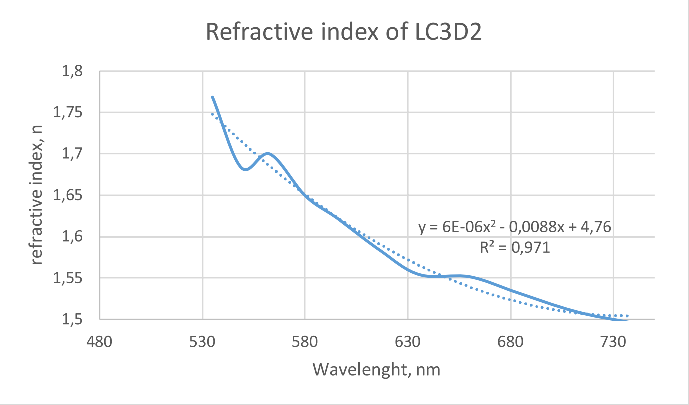


Fig. 5. Plotted *n* values of diffuser liquid crystal.

Refractive index measurement curve below 530 nm starts to have more interference, so 535 nm limit was used for curve estimation. The parabolic curve shape is in agreement with measurements for other LC mixtures in literature [6].

3. RESULTS AND DISCUSSION

To explain measured lower capacitance values than expected from calculations, we need to look at electro-optical response curves of the diffuser display, see Fig. 6. Before measurement, a diffuser display has static transmittance value between 30 % and 40 %, caused by relatively large light scattering domains that are orientated toward each other by weak intermolecular forces. Once low voltage is applied, transmittance drops to expected transient low value, as now previously large domains are broken down into smaller domains due to electric field which overcomes weak positional intermolecular forces. If the voltage is increased, LC molecules align in the direction of applied field.

Capacitance measurements can be only used when LC has reached uniform homeotropic alignment under electric field, and is not changing when field strength is increased. In this case, if 90 V with known capacitor in series is used, see Fig. 7, the measured value corresponds to simulated capacitance values. The LC layer thickness of 3.0 µm is larger than spacers being 2.7µm, used for LC layer thickness control, so we conclude that cells have little excess of LC volume and glass is not completely resting on spacers. Spectrometric results corelate with model calculations, so the model can be used for other LC layer thickness and size diffusers.

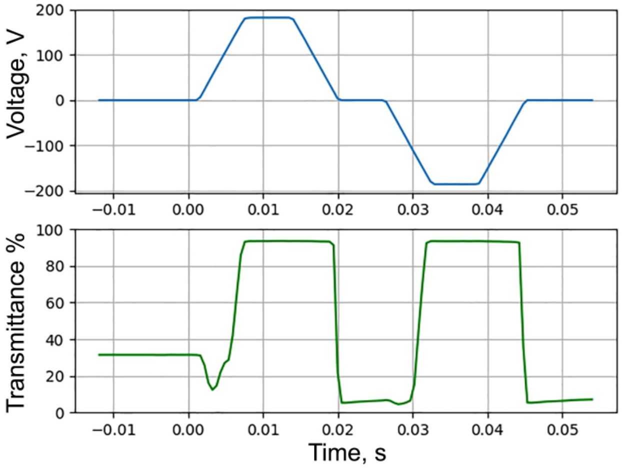


Fig. 6. Diffuser electro-optical response.

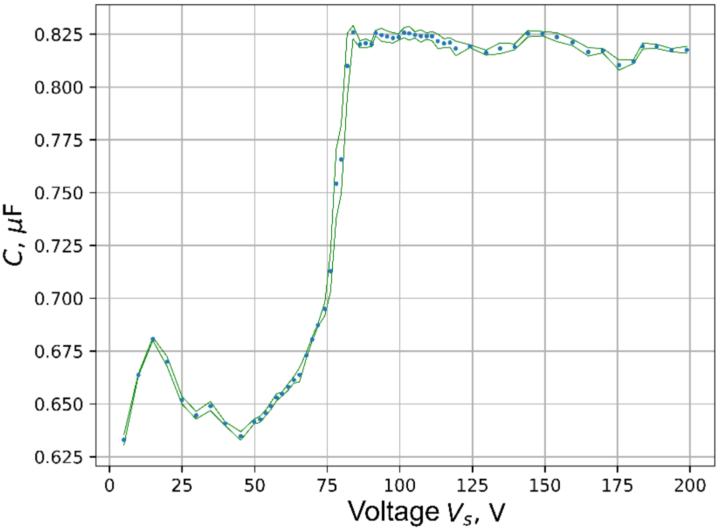


Fig. 7. Capacity measurements depending on measurement voltage with method “R”.

Four other diffusers of intended cell gap of 12µm (size 387.3x294.3mm) were selected for practical demonstration. All of them have quite identical current profiles during the charging and discharging processes.

For example, for the method with resistor, a graph of a resistor current starts as an approximately linear function and then transforms to an exponential decay curve for the second half of the charging process, see Fig. 8.

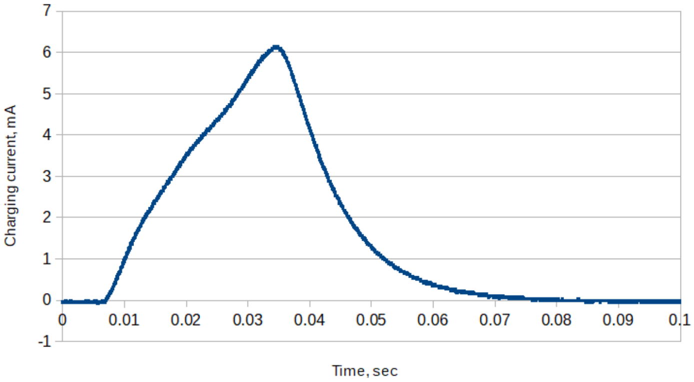


Fig. 8. Charging current of 387.3 x 294.3 mm 12µm diffuser in method "R".

This graph can be transformed to a function of a total charge that accumulates on a diffuser.

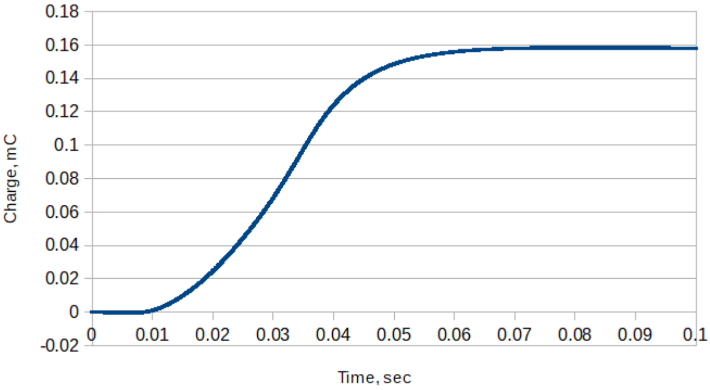


Fig. 9. Total charge stored on 387.3 x 294.3 mm 12µm diffuser during charging in method "R".

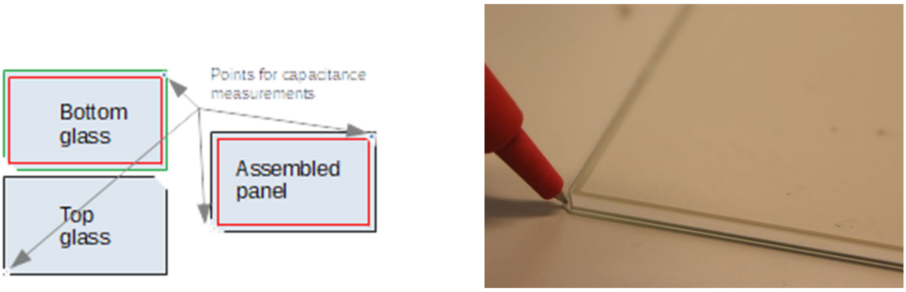
From Figs. 8 and 9, it is clear that 387.3 x 294.3 mm 12µm diffuser stores around 16 mC of charge; hence, by using Eq. (3), it is possible to conclude that its capacitance is equal to 0.8 µF. Approximately the same results were calculated for all of the diffusers in the same series, 4 in total. The consistency of the results indicates that that this capacitance value can be considered correct for this particular diffuser series and specific voltage supply level. With method C, we obtain the same capacitance values, as with method "R".

Table 5. Diffuser Capacitance Measurements

Display active area, mm	Calculated LC layer thickness, µm	Capacitance, Agilent 3606A, nF	Capacitance modelled, nF	Capacitance, method C, nF	Capacitance, method R, nF
61.8 x 48.8	3.0	50.8±2.9	70.2	69.7.±0.6	67±1
387.3 x 294.3	11.6	400	779.5	780±0.6	779±1

Using Comsol Multiphysics model, we find that the actual LC layer thickness is 11.6µ, see Table 5.

During the production process, it is important to measure LC layer thickness immediately after glass assembly into panel process, preferably by inline process control system. Coincidentally, standard display glass has notched corner for conductive side and flow direction identification purpose. After assembly both notched corners provide access to opposite display glass conductive layer, so capacitance value can be measured without completing further processing steps, like gasket curing, cutting and soldering of contact pads, see Fig. 10.



a) b)

Fig. 10. a) Assembled panel capacitance measurement points; b) close-up picture of panel corner during the measurement.

4. CONCLUSIONS

We have proposed the control method of scattering type liquid crystal layer thickness based on capacitance measurements. Various capacitance measurement methods have been evaluated and method with capacitor in series has been found most convienent. Comsol Multiphysics software has been used to compute capacitance models.

Capacitance method has been compared through a spectroscopic measurement method using interference spectrum peak detection for LC with a known refractive index, and it has been found that both provide similar results.

At the same time, diffuser LC was filled in identical LC layer thickness cells and previously obtained value of layer thickness was used to determine a refractive index of novel liquid crystal mixture for diffuser (light scattering) LC. Knowledge of refractive index allows for further optimization of LCD cells in future by index matching.

The proposed LC layer thickness control method has been approbed for use in inline LCD production.

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